

CLAIMS

1. Process for preparing high purity azithromycin comprising the following stages:
 - a) hydrogenating the iminoether (2) with Pt/C to obtain 9a-deoxo-9a-aza-9a-homoerythromycin A,
 - b) methylating the 9a-deoxo-9a-aza-9a-homoerythromycin A (3) originating from stage (a) with formaldehyde and formic acid,characterised in that the stage (a) is conducted in water to which acids have been added until a pH ≥ 4 is attained, the 9a-deoxo-9a-aza-9a-homoerythromycin A being isolated by crystallisation at the end of the reaction.
2. Process as claimed in claim 1, characterised in that the stage (a) is carried out after dissolving the iminoether at 5°C in water by adding an acid until a pH not less than about 4 is obtained.
3. Process as claimed in claim 2, characterised in that the pH is between about 4 and about 6.
4. Process as claimed in any one of claims 1-3, characterised in that said acid is phosphoric acid.
5. Process as claimed in any one of claims 1-5, characterised in that the stage (a) is conducted at a pressure between about 10 and about 40 bar.
6. Process as claimed in claim 5, characterised in that said pressure is between about 15 and about 25 bar.
7. Process as claimed in any one of claims 1-6, characterised in that the stage (a) is conducted at a temperature between about 0 and about 20°C.
8. Process as claimed in claim 7, characterised in that the stage (a) is conducted at a temperature between about 10 and about 15°C.
9. Process as claimed in any one of claims 1-8, characterised in that the separation of the crystalline form of 9a-deoxo-9a-aza-9a-homoerythromycin A by crystallisation is effected by a method which comprises the following stages:
 - i) the catalyst is eliminated by filtration and the reaction mixture is treated with an organic solvent immiscible with water and then with bases possibly dissolved in an aqueous solution, the product is extracted, and the solvent evaporated,
 - ii) the product originating from the preceding stage is dissolved in a solvent miscible with water, after which water is added in a quantity between about 1 and

about 100 volumes/volume of organic solvent at a temperature between about – 20 and +50°C, to obtain a suspension,

iii) the suspension is left under stirring for a time between 1 and 12 hours,

iv) the product is filtered off, washed with water and dried in an oven at about 40°C under vacuum at 40 mm Hg for 12 hours.

10. Process as claimed in claim 9, characterised in that in stage (i) of the crystallisation method, the base is chosen from NaOH, KOH, Na₂CO₃, K₂CO₃, ammonia and triethylamine.

11. Process as claimed in claim 9 or 10, characterised in that said organic solvent immiscible in water is chosen from the group consisting of cyclohexane, toluene, ethyl acetate, isopropyl acetate, ethyl ether, isopropyl ether, methyl tert-butylether, dichloromethane.

12. Process as claimed in any one of claims 9-11, characterised in that in stage (ii) acetone is used as the crystallisation solvent, the water being added to the extent of about 2 volumes/volume of acetone.

13. Process as claimed in any one of claims 9-12, characterised in that the temperature is between about 20 and about 25°C.

14. 9a-deoxo-9a-aza-9a-homoerythromycin A, in crystalline form, which under X-ray diffraction at the wavelength K α presents the image defined by the following table:

TABLE 1

Angle 2 θ	d (Å)	Relative intensity (I/I ₀)
7.285	12.125	100.0
11.290	7.831	57.5
12.595	7.022	64.9
14.590	6.066	58.0
18.405	4.817	61.0
19.320	4.590	40.2
21.005	4.226	32.3
22.355	3.974	35.0
22.800	3.897	38.3

29.630	3.762	31.7
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